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TECHNICAL MEMORANDUM WSRL-0303-TM

COLOUR TESTING OF NITROCELLULOSE PROPELLANTS BY A SPECTROPHOTOMETRIC METHOD

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TECHNICAL MEMORANDUM

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COLOUR TESTING OF NITROCELLULOSE PROPELLANTS BY A SPECTROPHOTOMETRIC METHOD

R.M. Kempson

SUMMARY

The colour test is a test for chemical stability that can be applied to many of the nitrocellulose propellants stabilized with ethyl centralite.

A spectrophotometric method has been developed for carrying out this test and for the preparation and checking of the associated standard colour solutions. Good agreement has been obtained with the method relying on visual comparisons. However, as visual comparisons of colour intensity are operator dependent and are more difficult at high intensities the spectrophotometric method is considered to be superior.

The satisfactory matching of results with those of Materials Research Laboratories for identical propellant samples has established consistency of colour number determinations following transfer of gun propellant surveillance work to Weapons Systems Research Laboratory.



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1. INTRODUCTION

An interlaboratory comparison of colour test results obtained by Materials Research Laboratories (MRL) and Weapons Systems Research Laboratory (WSRL) was carried out to establish consistency of results after the transfer of the test to WSRL. During the course of this work a spectrophotometric method was developed to replace the method relying on visual comparisons.

Propellants based on nitrocellulose (NC) are inherently unstable and slowly decompose from the day they are manufactured. Stabilizers are incorporated in the NC propellants to react with the nitrogen oxides liberated by the decomposition of the nitrate ester groups and therefore to greatly delay the onset of autocatalytic decomposition. After initial acceptance testing of propellants it is, however, necessary to carry out routine periodical inspections (RPI) in order to assess current stability and to estimate service life/safe life. The stability tests are applied to bulk propellant to assess serviceability prior to filling into ammunition and to propellants obtained from the breakdown of ammunition that is in Service use. There are many stability tests that have been developed for NC propellants but because there is no single test that on its own can give an acceptable evaluation of present or future stability it is usual to apply several tests to a particular propellant type.

RPI testing is currently carried out in Australia according to Army's OP 118(ref.1) and Navy's BR 1203(ref.2). The propellants are sentenced on the basis of the results obtained for the Abel heat test, the colour test (where applicable) and the residual stabilizer content.

The colour test is applicable to many of the double and triple base gun propellants that are stabilized with ethyl centralite (EC). When EC reacts with the nitrogen oxides it is converted to a number of yellow coloured nitro and nitroso derivatives. The colour test involves the comparison of an acetone solution of the propellant with standard ammonium cobalt sulphate/potassium dichromate colour solutions which results in the assignment of a colour number to the propellant. The colour number is an indication of the amount of EC that has been converted to its nitration derivatives and is therefore a simple means by which the chemical stability of the propellant can be judged. It is particularly suitable for a small test laboratory that does not have the facilities for carrying out the more complex stabilizer determinations.

The responsibility for gun propellant surveillance was transferred from MRL to WSRL in 1977. Since that time colour tests on propellants selected for RPI (Note 1) and on propellants undergoing the 49°C accelerated ageing trial have been carried out in this Laboratory. WSRL is also responsible for the preparation and checking of the colour standards and for their issue to other laboratories.

This memorandum gives details of the MRL - WSRL comparisons and the development of the spectrophotometric method for carrying out the colour test and preparation of the standard colour solutions.

Note 1. The Navy's laboratory at Kingswood is currently carrying out the RPI work on Navy's samples.

2. STANDARD COLOUR SOLUTIONS

Defence Standard 13-35(ref.3) (Note 2) describes the preparation of standard colour solutions Nos. 1, 2, 3, 4, 5, 10, 12, 15 and 20 by dilutions of the standard solution No.25. A Doboscq colorimeter is used to check that each solution has been prepared by the correct dilution factor and that there is continuity of the colour standards when a new batch of No.25 is prepared.

A simplified but more accurate procedure was employed for the preparation and checking of the solutions. It makes use of a spectrophotometer and the detailed procedure is described in Appendix I.

COLOUR TEST

3.1 Preparation of propellant solutions

A range of propellants to which the colour test is applicable was selected for testing. Most of the MNF2P/S and MNQF/S propellants had either been previously tested at MRL or had been transferred to WSRL for continuation of the 49° C trial. The remaining samples had been selected by Army or Navy for routine periodical inspection.

The propellant solutions were prepared in a similar manner to that described in BR 1203(2) (ref.2) and MQAD Method M85/77(ref.4). In the case of picrite propellants the undissolved picrite was allowed to settle out and an aliquot (20 mL) of the supernatant liquid was withdrawn and clarified by the addition of ethanol. For solutions that had stood for 3 days only 2 mL of ethanol were usually required, but if the solutions had stood for 2 days then 4 mL of ethanol were often required. The 3 day period was therefore used for all determinations.

Clarification was also attempted by centrifugation and by filtration through 0.45 μ m membrane filters, but as complete clarification was difficult to achieve the "settling-ethanol" method was retained.

The details of the preparation of the propellant solutions are given in Appendix I.

3.2 Colour number

In BR1203(2)(ref.5) the propellant solutions are compared with standard colour solutions in a Duboscq colorimeter. For solutions with colour numbers less than 5 the colorimeter may be dispensed with and comparisons are made in test tubes. In M85/77(ref.4) colour numbers are obtained by the use of a Lovibond 1000 comparator fitted with discs containing permanent colour standards. Colour numbers are quoted to the nearest whole number in both methods.

3.2.1 Visual determinations at MRL

While they were responsible for the colour test, MRL prepared the standard colour solutions in a similar way to Def Stan 13-35(ref.3) and obtained colour numbers of propellant solutions by comparisons in test tubes with a white background (ie without the aid of a colorimeter).

Note 2. Since the completion of the work described in this report MQAD Specification Circular No.9/82 has been received which advises that this standard has been withdrawn by MQAD and that reference should be made to MQAD Method M85/77 (see Section 3.2 and ref.4).

3.2.2 Visual determinations at WSRL

As neither the Duboscq nor the Lovibond colorimetry equipment (Section 3.2) was available the colour comparisons were done in test tubes illuminated by artificial light. Estimates of the colour numbers were made to a quarter of a colour number unit but this was only done with difficulty and it was now unusual for there to be a disagreement of 0.5 units between different operators. In cases where ethanol was added for clarification the colour number was corrected for the diluting effect of the ethanol. The colour numbers were then rounded off to whole numbers. In cases where comparisons were to be made with the spectrophotometric method (Section 3.2.4) the colour numbers were expressed to one decimal place.

The procedure for carrying out visual determinations is described in Appendix I.

Note: Most of the picrite propellants contain small amounts of dyes (typically 0.003%) for identification purposes: eg MNF2P/S pink, MNLF2P/M violet and MNQF/S yellow. The interference from non-yellow dyes was negligible but there was slight interference from the yellow dye in freshly manufactured propellant (but it was estimated that the colour number obtained for such a propellant was no more than 0.5 colour units too high). However all the dyes fade during storage of the propellants: this is only gradual for propellants stored at ambient but becomes very rapid (a few days or weeks) for samples undergoing elevated temperature trials. The outcome is that by the time a significant degree of degradation has occurred in a propellant the dye colour has completely faded and does not interfere at all.

3.2.3 Spectrophotometric determinations

The ultraviolet-visible spectrum of a standard colour solution was obtained on a Unicam SP700 spectrophotometer (figure 1). There were maxima at 260, 351 and 440 nm and a shallow trough before the 440 nm peak. As both acetone and major propellant ingredients absorb strongly in the UV, and as comparisons were to be made with visual determinations, only a visible wavelength could be used to measure colour numbers. A standard graph was therefore prepared at 440 nm using a Unicam SP500 spectrophotometer. A plot of absorbance against colour number (up to and including colour number 25) was linear: ie Beer's Law was obeyed. The plot up to colour number 10 is shown in figure 2.

The absorbance of each propellant solution was then measured at 440 nm, corrections were made for any ethanol added for clarification, and colour numbers were calculated to one decimal place. The effect of the addition of ethanol on the linearity of the absorbance versus colour number graph was checked by adding 4 mL of ethanol to 20 mL aliquots of acetone solutions of W and SC propellants (neither contains picrite). Absorbances were measured and colour numbers were calculated for the straight acetone solution and for the solutions diluted with ethanol (after correction for the dilution incurred). The two colour number values obtained for each propellant were identical (to one decimal place). This showed that when ethanol is added to clarify solutions of propellants containing picrite that the simple correction factor (see Appendix I) employed produces accurate results.

3.2.4 Comparison between the visual and spectrophotometric methods

The results obtained by the two methods were grouped under colour numbers in the ranges 0 to 1, 1 to 2, 2 to 3, 3 to 4, 4 to 5 and greater than 5. For each group average differences and the numbers of samples for which equal or higher results were obtained by the other method were calculated. The results are given in Table 1.

Considering the difficulty in obtaining visual estimations to fractions of a colour number it can be seen that the agreement between the two methods is very close. In none of the groups does one method yield results that are significantly different from those obtained by the other.

When a calibration graph was prepared at 445 nm (ie at a slightly longer wavelength than the maximum), and propellant solutions were measured at 445 nm the colour values were about 0.2 units less than those obtained at 440 nm. When similar determinations were carried out at 432 nm (ie about half way between the bottom of the trough and the maximum) the colour values were about 0.5 units higher than those obtained at 440 nm. The reason for this high dependence for the colour number that is obtained on the wavelength used for the determination can be seen from superimposing the visible spectra of propellant solutions on those of standard colour solutions (figures 3,4). The visible spectrum of a propellant solution is a smooth curve that rises steeply as the wavelength decreases and it will often cut across one or more of the spectra of the standard solutions.

To ensure that there is good agreement between the results obtained by the spectrophotometric and visual methods it is therefore important that the spectrophotometric measurements are performed at the maximum of the band in the visible (determined here as 440 nm).

3.2.5 Comparison between WSRL and MRL results

The WSRL spectrophotometric results were corrected for the dilution incurred during clarification, rounded off to whole numbers, and compared with MRL results (Table 2). All MRL colour test results obtained during the 49°C accelerated ageing trial of the propellants have been included so that the trends of increasing colour number with storage time can be demonstrated.

Except for a few cases where the WSRL result was either one unit higher or lower than the MRL result the colour numbers obtained were identical. For some samples there was no MRL result because the propellants had already been transferred to WSRL, but it can be seen that the WSRL values follow the usual trend.

4. DISCUSSION

Unaided visual determinations are operator dependent but are generally adequate for colour numbers up to 5 and are considered suitable for small test laboratories operated by the Services that do not possess other equipment.

The use of a colorimeter would improve the accuracy of visual determinations, and could probably be used for colour numbers greater than 5. However the use of a spectrophotometer removes any operator dependence associated with judging colour intensity and allows colour numbers to be accurately quoted to one decimal place (if required) including those much greater than 5. The spectrophotometric method is also far superior to visual methods for the preparation and checking of the standard colour solutions.

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The colour number values obtained by the spectrophotometric method depend on the wavelength used for the determinations. It is therefore important that the instrument is correctly calibrated for wavelength and that measurements are carried out at the maximum of the band in the visible. Good agreement was obtained with the visual method under these conditions.

5. CONCLUSIONS

The spectrophotometric method is superior to visual methods for the preparation and checking of standard colour solutions and for carrying out the colour test on propellants.

Good agreement has been obtained between MRL and WSRL for the colour test of picrite propellants.

6. ACKNOWLEDGEMENTS

Thanks are extended to Mr R.G. Davidson of MRL for supplying many of the propellant samples and to Mr D.J. Pinson of MRL for supplying the MRL colour test results.

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No.	Author	Title
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2		Instructions for the Quality Assurance of Naval Armament Stores, BR1203 Series 2 UK Ministry of Defence, November 1971
3		"Standard Colour Solutions for Propellant Testing". Defence Standard 13-35, Ministry of Defence, London, 1968
4		"The Colour Test of Colloidal Propellants". Laboratory Method M85/77, Ministry of Defence, London, March 1977
5		"The Colour Test of Propellants". Ref.2 Chapter 17 Section D

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TABLE 1. A COMPARISON OF THE VISUAL AND SPECTROPHOTOMETRIC METHODS

Pı	Propellant		Colour No.		Average	Number of Samples		
Туре	Lot No.	Sample*	Visual	Spectro- photometric	Difference (regardless of sign)	Identical Colour No.	Result	Spec/ic Result Higher
MNF2P/S MNF2P/S MNF2P/S MNQF/S NQ/M NQ/M	MDK770	Initial Initial Initial Initial RPI RPI	0.3 0.4 0.3 0.8 0.8	0.3 0.5 0.5 0.9 0.7	0.1	1	2	3
MNF/S SC048 NQ/S HSCK/T MNQF/S	MEC425 MEC159 MEC231 RNC5776 MEC770	RPI RPI RPI RPI Initial	1.1 2.0 1.4 1.7	0.8 1.9 1.1 1.6 0.9	0.2	0	5	0
MNF2P/S MNF2P/S MNF2P/S MNQF/S	MDK762R MDK763 MDK766	36 24 36 24 24 24 24 RPI RPI	2.7 2.2 2.7 2.2 2.2 2.7 2.4 2.7 2.8	2.9 2.5 2.9 2.2 2.6 2.6 3.0 2.4 3.2	0.3	1	2	6
	MDK732 MDK733 MDK735 MDK737 MDK748 MDK750 MDK751	48 48 48 48 48 36 36 36 36 48 36 RPI	3.3 3.3 3.3 3.9 3.6 3.0 3.3 3.8 3.9 3.3	4.0 3.6 3.4 4.0 3.8 3.1 3.1 3.5 4.0 3.0 3.4	0.2	0	3	9
MNF2P/S MNF2P/S MNF2P/S MNF2P/S MNF2P/S MNF2P/S MNQF/S SC103	MDK736 MDK738 MDK753 MDK764	48 48 48 36 36 48 60 RP	5.2 4.7 4.4 4.5 4.7 4.1 4.4	4.5 4.7 4.3 4.4 5.1 4.0 4.8 4.2	0.2	1	4	3
	MDK761 MDK762	48 60 48 60 48 36	> 5 > 5 > 5 > 5 > 5 > 5 > 5 > 5	5.6 5.7 6.5 5.7 6.0 5.8 6.9	1	Not applica	able	

Initial = sample as received

12, 24, 36, 48, 60 = number of months on 49°C trial

RPI = sample for routine periodic inspection

TABLE 2. CONTINUITY OF RESULTS FROM MRL TO WSRL

Propellant		Colour Numbers		Propellant		Colour		
Type/Lot No.	Storage at 49°C	(corrected)		Type/Lot No.	Storage at 49°C		Numbers (corrected)	
	(months)	MRL	WSRL		(months)	MRL	WSRL	
MNF2P/S				MNF2P/S				
MDK719	0 12 24 36 48 60	0 2 3 NS 4 5	6	MD K7 37	0 12 24 36 48 60	1 2 3 4 4 4	4	
MD K729	0 12 24 36 48 60	1 2 NS 3 4 5	4	MDK738	0 12 24 36 48 60	<pre></pre>	4	
MDK731	0 12 24 36 48 60	0 2 NS 3 4	4	MDK745	0 12 24 36 48 60	< 1 NS 2 3 3 4	3	
MDK732	0 12 24 36 48 60	< 1 2 3 3 4 4	4	MDK748	0 12 24 36 48 60	< 1 2 3 4 4 5	4	
MDK733	0 12 24 36 48 60	< 1 2 3 4 4 4	3	MDK750	0 12 24 36 48 60	<pre></pre>	3	
MDK735	0 12 24 36 48	1 2 3 3 4	3	MDK751	0 12 24 36 48 60	<pre></pre>	3	
MDK736	0 12 24 36 48 60	< 1 2 3 3 4 5	5	MDK753	0 12 24 36 48 60	< 1 2 3 4 5 5	4	

TABLE 2(CONTD.).

Propel1	ant	Cold	our	Propel1	ant	Colour	
Type/Lot No.	Storage at 49°C	Numbers (corrected)		Type/Lot No.	Storage at 49 C	Numb (corre	
	(months)	MRL	WSRL		at 49 C (months)	MRL	WSRL
MNF2P/S				MNQF/S			
MDK762R	0 12 24 36 48	0 2	2 4 4	MEC 777	0 12 24 36 48	0 2 3 NS 4	
MDK763	0 12 24 36 48 60	< 1 1 2	3 4 4	MEC 79 5	60 0 12 24 36 48	1 2 3 3 4	5
MDK764	0 12 24 36 48 60	0 2 4	5 6 7	MEC822	60 0 12 24 36 48 60	4 0 NS 2 3 3	3
MDK765 MDK766	0 12 24 36 48 60	0 2 3	3 5 6	MEC 823	0 12 24 36 48 60	1 NS 4 > 5 > 5 > 5	7
MDK768	12 24 36	0 2	2 3	MEC 824	0 12 24 36	1 2 3 4	3
EDN 700	12 24 36 48	NS	3 4 5	MEC835	0 12 24 36	< 1 2	3 4 5
MDK797	0 12 24 36 48 60	<pre> < 1 2 NS 3 4 5</pre>	4		48		5

NOTE 1 MRL results were obtained visually

WSRL results were obtained by the spectrophotometric method and rounded off to whole numbers

NOTE 2 NS = Not sampled

APPENDIX I

THE DETERMINATION OF THE COLOUR NUMBER OF PROPELLANTS (VISUAL AND SPECTROPHOTOMETRIC METHODS) AND THE PREPARATION OF THE REQUIRED COLOUR STANDARDS

I.1 Principle

The colour test is a rapid and simple way of assessing the chemical stability of many of the nitrocellulose propellants that are stabilized with ethyl centralite (eg the "picrite" and "SC" type propellants). The colour intensity of an acetone solution of the propellant is compared with standard ammonium cobalt sulphate/potassium dichromate solutions, either visually or spectrophotometrically, and the propellant is assigned a colour number.

I.2 Reagents

- I.2.1 Potassium dichromate: analytical reagent quality grind to a fine powder and dry to a constant mass at 140-150°C.
- I 2.2 Ammonium cobalt sulphate hexahydrate: containing not more than 0.003% iron and 0.01% nickel, assay (ex cobalt) $100 \pm 2\%$. BDH product No. 27156 meets these requirements.
- I.2.3 Sulphuric acid: analytical reagent quality RD 1.84.
- I.2.4 Acetone: analytical reagent quality.
- I.2.5 Ethanol: 95% v/v.

I.3 Apparatus

- I.3.1 Test tubes (to be used for visual determinations of colour number): with 14/19 ground glass joint, nominal dimensions length 140 mm, internal diameter 15 mm, external diameter 18 mm.
- I.3.2 Conical flasks: 100 mL with rubber stoppers covered with aluminium foil.
- I.3.3 Spectrophotometer: capable of measuring absorbance at 440 nm and being read to 0.001 absorbance units at 0.5 absorbance.
- I.3.4 Cells: matched, silica or glass, path length 10 mm.

I.4 Procedure

- I.4.1 Preparation of standard colour solutions
 - I.4.1.1 Prepare standard colour solution No.25 by dissolving exactly 1.210 \pm 0.001 g of potassium dichromate, 32.70 \pm 0.01 g of ammonium cobalt sulphate hexahydrate and 12.5 \pm 0.2 mL of sulphuric acid in water. Make up to 1 L in a volumetric flask.
 - I.4.1.2 Prepare standard colour solutions Nos. 1, 2, 3, 4, 5, 10, 12, 15 and 20 by pipetting 4, 8, 12, 16, 20, 40, 48, 60 and 80 mL respectively of the standard colour solution No.25 into 100 mL volumetric flasks and making up with water. Store the solutions in the dark when not in use.

- I.4.1.3 Measure the absorbance of each standard colour solution in the 10 mm cells at 440 nm using water in the reference cell. Plot absorbance against colour number and draw in the straight line of best fit. If there is any point that does not lie close to the line then check its absorbance and, if necessary, repeat the dilution. Check all solutions every six months and prepare fresh solutions if necessary.
- I.4.1.4 If visual determinations of the colour number are to be made fill the test tubes with the standard solutions, insert 14/19 ground glass stoppers, and number each tube clearly with the number of the standard solution that it contains. Fill a test tube with distilled water to act as colour number 0.
- I.4.2 Preparation of propellant solution
 - I.4.2.1 Carry out the colour test on each propellant sample in duplicate.
 - I.4.2.2 Grind the propellant to be tested so that all the ground sample passes a sieve of 2 mm aperture. Transfer 50 mL of acetone by pipette to a conical flask and add 1.00 \pm 0.01 g of the ground propellant while swirling the flask. Stopper the flask securely with a rubber bung covered with aluminium foil and keep the flask out of any direct light at all times. Swirl the flask occasionally until no more material will dissolve.
 - I.4.2.3 For propellants that do not contain picrite allow the flask to stand overnight: if the sample contains any chalk it will have settled out. Transfer 20 mL of the solution by pipette into another 100 mL conical flask and stopper.
 - I.4.2.4 For propellants that contain picrite keep the flask in the dark and allow contents to remain undisturbed for three days to allow as much as possible of the insoluble portion to settle out. Without disturbing the sediment carefully transfer 20 mL of the supernatant liquid by pipette to another 100 mL flask. Add 2 mL of ethanol by pipette to the second flask and shake vigorously until the solution is completely clear of any turbidity. A further 2 mL of ethanol may be added if complete clarification was not achieved. If clarification is still not achieved then too much picrite has been transferred and the determination must be repeated.
- I.4.3 Visual determination of the colour number

This method is suitable for the determination of colour numbers up to 5. Unit intervals above 5 are too difficult to judge visually and the spectrophotometric method should be used in these cases.

I.4.3.1 Fill a test tube with the propellant solution to be tested and stopper with a rubber stopper covered with aluminium foil. Match the colour of the propellant solution against the standard colour solutions either by inclining the test tubes against a white background or by placing them on a translucent Perspex screen illuminated from behind. It is important that all the tubes are equally illuminated and some experimentation will be required to determine the best method of viewing the tubes. If ethanol was not added estimate the colour number to the nearest standard. If ethanol was added attempt to estimate the colour number (uncorrected) to the nearest 0.25 of a colour number: if this is not possible then estimate to the nearest 0.50.

- I.4.4 Spectrophotometric determination of the colour number
 - I.4.4.1 Measure the absorbance of each standard colour solution at 440 nm, plot absorbance against colour number units, draw in the line of best fit, and calculate the slope of the graph (m).
 - I.4.4.2 Fill the sample cell with the propellant solution and measure the absorbance at 440 nm using water as the reference.
- I.5 Expression of results
 - I.5.1 Visual
 - I.5.1.1 If ethanol was not required for clarification quote the colour number as that of the nearest matching standard.
 - I.5.1.2 If ethanol was required a correction is made for the dilution incurred.

Colour number = Colour number (uncorrected)* x $\frac{(20 + v)}{20}$

Where v = volume of ethanol (either 2 or 4 mL) required for the clarification of 20 mL of propellant solution. Quote the result to the nearest whole number.

- * Estimated to 0.25 or 0.50 units paragraph I.4.3.1.
- I.5.2 Spectrophotometric method

Colour number = $\frac{A}{m} \times \frac{(20 + v)}{20}$

Where v = as in paragraph I.5.1.2

A = absorbance at 440 nm

m = slope of standard graph in absorbance units per colour number.

By this method the result may be quoted to one decimal place if required.

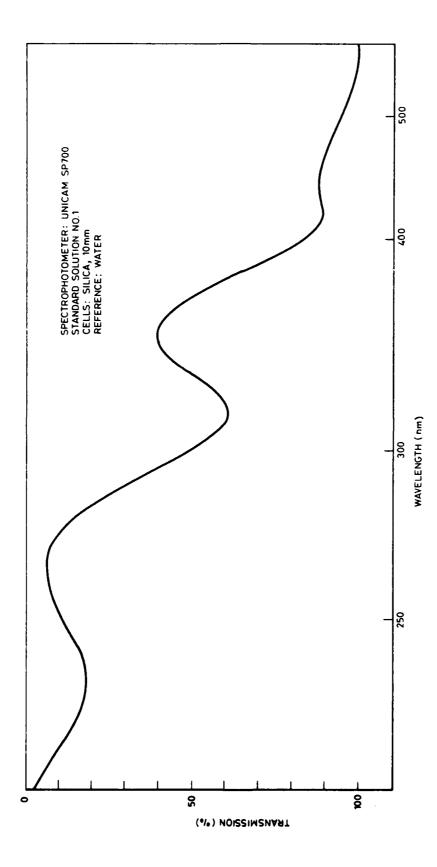


Figure 1. Ultraviolet - visible spectrum of a standard colour solution

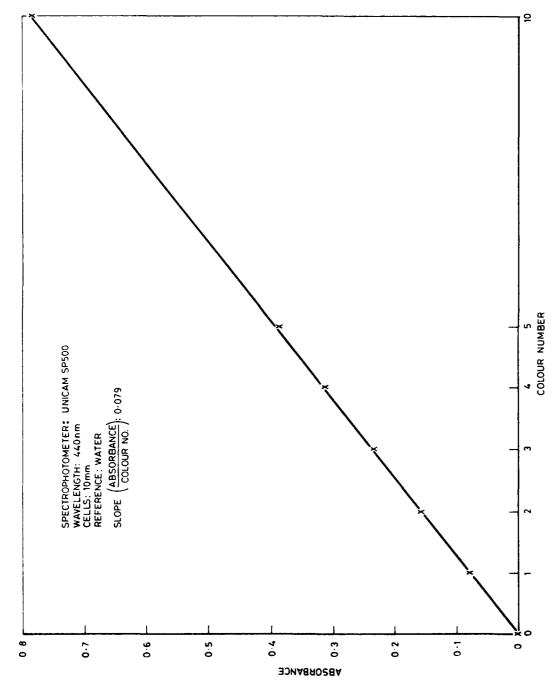


Figure 2. Absorbance - colour number plot for standard colour solutions

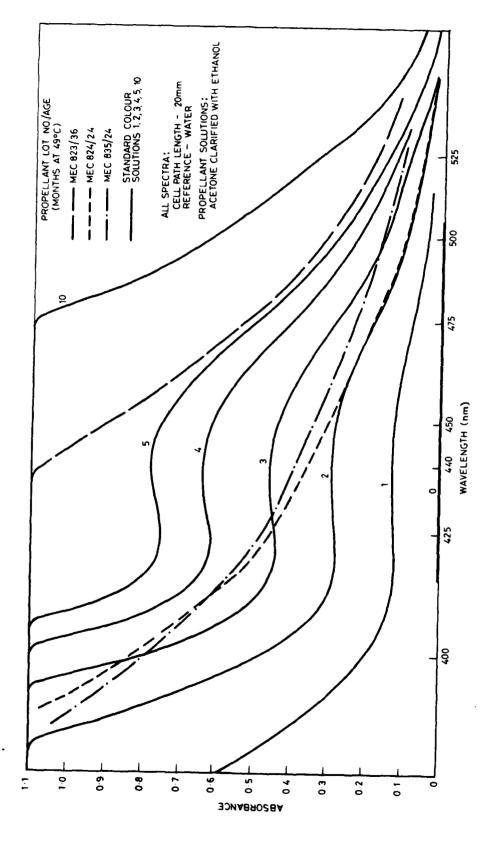


Figure 3. Visible spectra of solutions of propellant type MNQF/S

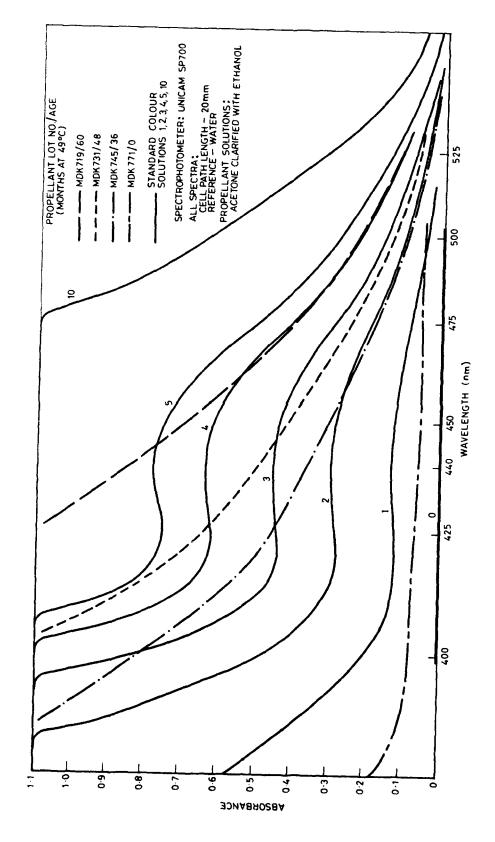


Figure 4. Visible spectra of solutions of propellant type MNF2P/S

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16 SUMMARY OR ABSTRACT:

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The colour test is a test for chemical stability that can be applied to many of the nitrocellulose propellants stabilized with ethyl centralite.

A spectrophotometric method has been developed for carrying out this test and for the preparation and checking of the associated standard colour solutions. Good agreement has been obtained with the method relying on visual comparisons. However, as visual comparisons of colour intensity are operator dependent and are more difficult at high intensities the spectrophotometric method is considered to be superior.

The satisfactory matching of results with those of Materials Research Laboratories for identical propellant samples has established consistency of colour number determinations following transfer of gun propellant surveillance work to Weapons Systems Research Laboratory.

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